“Studies on Effects of Shock wave impacts on Structural changes of Poly(vinyl alcohol)”

Synopsis of the doctoral thesis in Physics to be submitted to

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June 2022

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Title	"Studies on Effects of Shock wave impacts on Structural changes of Poly(vinyl alcohol)"
Date of Registration and References	**07.01.2013** 1: MU/EXB/Ph.D/CR.21/Phy./2014-15/E.52 dated 15.12.2014 2: MU/ACC/Ph.D/CG/CR-14/2016-17/A3 dated 27/06/2017 3: MU/EXB/Ph.D/CR.20/Phy./2014-15/E.13/E.25 Dated 16/09/2021 4: MU/EXB/Ph.D/CR.20/Phy./2014-15/E.13/E.25 Dated 07/12/2021

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1. Introduction

Shock waves are the wave instances, produced when a sudden burst takes place in a fluid medium. These waves manifests as a sudden increase in the pressure, temperature and enthalpy of the medium for a very short duration [1-3]. The phenomenon of shock waves is commonly associated with aerospace engineering/aeronautics and in particular with supersonic flight. Shock waves appear in nature whenever the different elements in a fluid approach one another with a velocity higher than the local speed of sound. Dissipation of energy, rapid changes in velocity, pressure, temperature and flow turning are some of the features associated with shock waves [4-5]. The impact of shock waves on any surface is a momentary effect. But, during which, it may create sustainable changes due to very high pressure, temperature and other physical conditions. The capability of shock waves to generate non-linear pressure and temperature spikes in the medium of propagation, finds very interesting applications in variety of fields such as medicine [6,7,8], biological sciences [8,9,10], material processing [11-28], manufacturing and other industries [3,10,29]. Therefore, it is very much required to produce shock waves in the laboratory in a controlled manner. Among the different methods of production of shock waves, shock tubes or shock tunnels are most popular.

Shock tube or a shock tunnel is an instrument used to produce shock waves in laboratory in a controlled manner [29-33]. Recent works of Reddy et all [32,33], demonstrated a miniaturized shock tube, capable of producing low magnitude shock waves, which is the main motivation for this work. Many institutions and researchers across the country are using this device for academic and research works.

Generally, a shock tube consists of a driver section, a driven section, and a thin diaphragm separating the two [30,31]. When a high pressure is created by any means of compression of the medium in the driver section, the diaphragm ruptures by producing a shock wave in the driven section. The shock produced will move with a supersonic speed which is measured in terms of 'Mach numbers'. Mach number is the ratio of the speed of the object in a medium to the speed of the sound in the medium [3]. Thus Mach 1 refers to the speed of sound in a medium and more will represent multiples of the speed of the sound. The supersonic speed shock wave data is extracted from the high frequency piezoelectric pressure and temperature sensors. The Mach number of the

shock wave generated can be calculated from the output plots of the data recorded on a high speed data logger. The diaphragm rupture pressure can be noted from the pressure gauge attached. With this data the other parameters like pressure and temperature jump at the primary shock wave front can be calculated using normal shock relations or R-H equations [2, 3].

Thus, the proper analysis of the data obtained from any free-piston or compressed-gas driven shock tube or a shock-tunnel needs a high precision oscilloscope with data logging facility. Further, processing of this huge data and getting the required output takes few minutes to sometimes few hours of human effort. Also, the cost of these devices limits the usage of shock tubes in small budget research projects. The work presented here also involves development of a cost effective technique which provides a state-of-the-art data acquisition and processing facility for a shock tube or tunnel.

Recently, shock wave applications have increased in all domains, and the shock wave treatment on materials for the search of novel properties is gaining popularity among the shock wave researchers [11-29]. There are a quite number of references shows the significant effect on the structural and chemical property changes by shock waves on nano materials, nano particle composites, metal composites, ceramics etc. [13-28]. Also, noticeable changes reported on optical, electrical and dielectric properties of materials [24-27] by shock waves. But there is no much work carried on the effects of shock waves on polymers except one reference on polyurethane [28]. Therefore, there is a lot of scope for doing research in this domain. So, we found interesting and worthwhile in working on shock wave treatment on a polymer material. So, we decided to work on Poly(vinyl alcohol), commonly known as PVA, which is easily available, environmental friendly and relatively has more applications.

Poly Vinyl Alcohol (PVA) is a water-soluble biopolymer, with carbon chain backbone and hydroxyl (OH) groups attached to methane carbons. These OH groups are the source of hydrogen bonding and therefore responsible for the formation of polymer [34]. PVA has many interesting physical and chemical properties such as good mechanical strength, water soluble, nontoxic, biodegradable, high dielectric constant etc [35]. Therefore, PVA finds many applications in diverse fields. With different kinds of

additives or dopants, these properties can be further improved or fine-tuned [34 - 38]. Also, there are fair numbers of irradiation (electron, gamma, UV etc.) techniques applied to modify the properties of PVA films [39 - 42]. The work presented here is a novel method to process PVA or any material in general using shock waves. The work focused mainly on the structural and electrical properties of PVA. Because, it is well established that, including PVA polymers are good insulators. Therefore, any attempt to increase the conducting property of polymers finds much more applications in electrical and electronic industry. So, we got motivated in attempting to increase the conducting property of PVA along with the other interesting properties using shock wave treatment.

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2. Aim and objectives of the present study:

In the present study an in-house developed shock tube facility was setup and the same is used to study the effects of shock wave impacts on the PVA films. The shock wave treated PVA films were tested for physical, chemical and electrical property changes. The gamma irradiation effects were also carried out on the shock treated PVA films and studied for the physical, chemical and electrical property changes.

The main objectives of the study are as follows:

1. Development and setup of an in-house shock tube facility at the working place.
2. Calibration and validation of the shock tube developed.
3. Shock wave treatment of PVA thin films with varying number of shock wave impacts.
4. Study of physical and morphological changes of PVA due to shock wave impacts.
5. Study of chemical composition changes of PVA due to shock wave impacts.
6. Study of electrical properties changes of PVA due to shock wave treatment.
7. Study of effect of gamma irradiation on the shock wave treated PVA films.

3. Overview of research work:

An instrument called shock tube which is capable of producing shock waves with the burst of a paper/polythene diaphragm is designed and developed. The developed instrument is tested for accuracy and consistent results. The device is calibrated in comparison with the theoretical values.

The PVA thin films were prepared using casting evaporation technique. The prepared samples were placed at the open end of the driven section of the shock tube at a distance of around 1 cm. The shock waves are generated by the burst of a 120GSM art paper used as the diaphragms in the shock tube. The process is repeated to get continuous shock waves by replacing the diaphragms in a time interval of around 15 seconds. The samples were treated with shock waves of 0, 25, 50, 100 and 150 impacts. The PVA films were peeled off carefully from the petri-dishes after the treatment with shock wave for further studies/characterization.

The shock wave treated PVA films were characterized using Fourier Transform Infrared Spectroscopy (FTIR), Ultraviolet-Visible Spectroscopy (UV-Vis), X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), and Energy Dispersive Spectroscopy (EDX). The Contact angle measurement and water solubility test were conducted to know the hydrophilic and hydrophobic behavior of the shock wave treated PVA films.

Further, shock wave treated PVA films were gamma irradiated to test the morphological, chemical and electrical property changes.

The treatment of materials with shock waves is a novel method of material processing and there are very few literatures available to quote in this area of work. The method proposed and used here is an indigenous technique of material processing. The results obtained are also unique and are very promising for future developments of material processing.

4. Outline of the Research Work:

The research work carried out is divided into six different chapters. The contents of these chapters are listed below:

4.1 Chapter I: Introduction and Literature Survey

Chapter-I gives a comprehensive detail about shock waves, shock wave production techniques/tools - shock tubes, applications of shock waves along with the material under study - PVA structure and properties. The traditional methods used for the shock waves production, advantages and disadvantages are discussed. Extensive literature survey has been carried out on all the above aspects. The advancement of shock wave technology and their applications in diverse fields in the recent years have been discussed. Importance of shock wave treatment on materials and their applications are also discussed in detail. Scope of research in the field of material processing by means of shock wave impacts has no limit and is an emerging field of research. So, thorough studies have been covered and surveyed in literature review. The plan of present research work was derived based on the literature review.

4.2 Chapter II: Materials and methods

The chapter II contains the description of the preparation of the PVA films and the method of shock wave treatment on the sample films. The details of the instrument used for the shock wave treatment ie., the shock tube. All the characterization techniques used in the present study. Namely, the Fourier Transform Infra-Red (FTIR) spectroscopy, Ultra-Violet and Visible spectrum spectroscopy, The X-Ray diffraction studies, Field Effect Scanning Electron Microscopy (FESEM), Energy Dispersive X-Ray (EDX) analysis, AC and DC conductive studies, Dielectric and impedance analysis, Contact angle and water solubility tests. The theoretical background, usage methods and the instruments used for all the above techniques are discussed in detail.

4.3 Chapter III: Design and Development of Shock tube setup

This chapter describes the design, development and setting up of shock wave experimental setup. As explained in the chapter-I, the need of an alternative cost-

effective facility to work on shock waves is discussed here. The shock tube developed is a modified and cost effective device; completely designed, fabricated and programmed in-house and the same is installed at the working place. A 'Shock tube Data Processor' device of 5 MHz speed is developed using the open-source hardware platform 'Arduino' and a 'Shock tube Data Analyser' software is developed using the open source software development platform 'PROCESSING'. Thus, the shock tube developed is equipped with an indigenous shock wave data processing device and supporting software. The detailed process of development and the necessary technical details like electronic circuitry and software and hardware programming is described in detail. The in-house developed shock tube not only reduces the cost of the device, technically more efficient than the counterparts. The cost effectiveness is achieved by using open-source hardware and software platforms [43-46]. In this chapter the detailed description of all the technical aspects as well as the working of the shock tube is discussed. Also, the testing and calibration procedure of the device is explained along with the theoretical calculations and comparison with the standard model.

4.4 Effects of Shock wave treatment on Poly(vinyl alcohol)-PVA films

After the PVA films were treated with shock waves it is found that there are significant changes in the physical, chemical and electrical properties. The possible mechano-chemical effects by which the shock waves may create these changes are discussed in detail. These changes were confirmed by characterizing the samples with well-known techniques. This chapter comprises with all the results of the material characterizations and detailed discussions of their interpretations.

Physical and chemical property studies:

FTIR spectroscopy was used to assess the functional groups involved in the PVA treated with shock waves. Alterations in the important peaks of FTIR spectra clearly support the successful modification in PVA functional groups by shock waves. The spectrum shows some dominating peaks related to the hydroxyl and acetate groups. A broad band observed between ~3600 to ~3200 cm^{-1} which is associated with O-H stretching from intermolecular and intra-molecular hydrogen bonding. There is a

reduction in the O-H stretching peak after shock waves treatment comparing with the pristine PVA samples situated at 3300 cm^{-1}. This reduction in hydrogen bonding indicates that the molecules becoming less polar. This means that molecules are less soluble in a polar solvent such as water which indirectly supporting that the material becoming less water soluble after shock waves treatment [47]. The sharp vibrational peak at 2900 and 1700 cm^{-1} is attributed to the C-H asymmetric stretch from alkyl groups and C=O stretching respectively.

The careful observation of UV-Vis spectra revealed that, the wavelength of absorption spectrum shifted towards higher wavelength regions from untreated PVA sample to higher number shock waves treated samples. This trend is confirmed by examining the wavelengths corresponding to the highest absorbance value for different colored curves employed to indicate untreated and treated with different shock numbers. The treatment of PVA samples with shock waves brings systematic modification to its host atomic/molecular environment for electron confinement. These modifications would be mainly of two types named as (a) chain session and (b) crosslinking [48, 49]. Breaking of long polymer molecules after shock wave treatments is clearly depicted in the SEM images. The rearrangement / breaking / cross linking of PVA molecules due to the shock waves, increases the delocalization in the polymer bonds. This increase in delocalization lowers the energy gap between the highest occupied molecular orbital and the lowest unoccupied anti-bonding orbital. It needs less energy to make the jump and so a longer wavelength of light is absorbed. Thus, as the absorptions move to longer wavelengths the amount of delocalization in the molecule increases [47]. This clearly says that the electron confinement of the samples was modified to exhibit higher delocalization with respect to shock wave treatment.

The surface morphology of the shock wave treated PVA films were studied with FESEM analysis. Broadly it can be observed that the shock wave treated samples with 100 and 150 shots have changed their morphology after the treatment. The untreated sample show a stable plane surface morphology of the PVA films with no distortions or cracks. The interaction of shock waves with polymers breaks the long chain molecules and lead for cross linking of them. Such breaking and cross linking of molecules lead to increase of carbon elements per unit volume. This hypothesis is clearly confirmed in the

EDX spectra as well. The increment of carbon concentration is evidenced from the increase of carbon peak for the modified samples.

Electrical property studies:

DC electrical conductivity studies support the aspects on delocalization of electrons discussed in the UV – Vis results after shock waves treatments. The shock wave treated samples have shown significant increment of DC electrical conductivity due to increase of such delocalization process of electron. Normally, PVA is an insulating material and non-conducting polymer. The untreated samples of them would exhibit lower electrical conductivity for alternating or direct current. However, treated samples showed increase in the rate of current for the same voltage values when DC was applied. This indicates the decrease in the resistivity of the samples due to shock wave treatment and thereby increasing their conductivity. The only possible way of such increment of conductivity trend of an insulating material is due to change in its path of charge conducting environment. This conductivity is due to carbonization of the material due to shock wave treatment where breakage of some bonds of the polymer chain pull number of carbon atoms towards each other which would facilitate for conduction. However, this is not the only possible way which has leaded the PVA to exhibit such increment in the conductivity trend. There may be some debris of polymers created with some charge on them when shock waves hit the polymer samples. These debris or free radicals move with respect to their opposite polarity when direct current is applied to partially contribute to reduce the higher resistive region.

The further evidence on the increase in conductivity was obtained from the impedance analysis for the AC voltages. Cole-Cole plot of the treated samples in the range of 40 Hz to 5 MHz frequency sweeping, is studied. Decrement in the half circle of the plots for treated samples with respect to untreated PVA samples indicates the increase of the overall conductivity of the samples.

XRD Analysis

X-ray crystallographic studies were carried out to test the possible changes in the physical properties of the shock wave treated PVA samples. The X=Ray diffraction

studies revealed that there is a significant increase in the crystallinity of the PVA samples after the shock wave treatment. From the XRD pattern it is found that there is a remarkable increase in the crystallinity of the samples. This increment is proportional to the increase in the number of shock wave impacts. This is in good agreement with the concept of stress induced crystallinity of polymer materials [48, 49].

Water solubility test and contact angle measurement:

From the FTIR and UV-Vis analysis it is observed that the shock wave treated PVA samples becoming less water soluble. To confirm the observation an easy and direct measurement technique was used to test the water solubility of the samples. The test was performed against shock waves for PVA samples of 0.4mm thick films. The samples were folded to a cone shape and placed in a conical glass flask. 1 ml of water is poured into it and at the same time a stop clock was turned on. The time taken for the first drop of water to fall into the conical flask after dissolving through the PVA films was recorded. As expected, the water drop from the shock wave treated sample fell into the conical flask with a delayed time compared to untreated PVA sample. The test was repeated with the same number of shocks for two more samples and the similar results were found. Therefore, after treatment with shock waves, it supported the claim that the PVA samples were moved to less water solubility.

Further, water contact angle of pristine PVA and shock treated PVA samples were measured in order to evaluate the hydrophilic nature of the material surfaces. In good agreement with the previous results [38], the neat PVA had a very low water contact angle of 24.8°. Whereas, there is a remarkable increased water contact angle by 85° for shock wave treated films compared with that of neat PVA counterpart. Thus, the shock treated samples shifted from hydrophilic nature to the hydrophobic. This remarkable hydrophobic improvement for PVA films could be associated with the FTIR studies that the reduced hydroxyl groups in PVA with the shock treatment.

4.5 Chapter V: The gamma irradiation studies on the shock waves treated PVA films

The shock wave treated PVA films were irradiated with gamma rays for a dose of 100 kGy, to test the irradiation effects on the samples. It is found that, after gamma

irradiation in the shock wave treated samples, there is a remarkable increase in the crystallinity of the samples at 2θ=16.26 degrees. The increase in the crystallinity of the samples is linearly dependent on the number of shock waves impacts on the sample films. This may be attributed to the strain-induced crystallinity with radiation [50]. After the gamma irradiation on the shock waves treated polymers, the material electrical properties started to resume. This is due to, the gamma irradiation can penetrate into the polymer materials and may relax the molecular strain induced by shock waves. Therefore, internal annealing of the material might have been taken place. The cole-cole plot also depicted the decrease of conductivity by increasing the curvature towards higher frequencies after gamma irradiation. This is implying that the earlier (before shock wave treatment) electrical properties were regained after gamma irradiation.

4.6 Chapter VI: Summary and Conclusion

This chapter concludes all the findings of research work carried out in the earlier chapters. It is clear from the observations of present study, that the shock wave treatment on PVA films has significantly altered the physical and chemical properties of the material. The FTIR study shows the reduction in the O-H groups of the PVA molecules which lead to the reduction in the water solubility of the material. The UV-Vis analysis confirms the significant shift in the absorption wavelength due to the chain session and delocalization of main PVA chains. The FESEM analyses confirm the changes in the surface morphology. Significant increase in the conductivity was observed with respect to shock wave treatment. The studies show that the shock wave treatment can be used as a tool for processing of materials. Increment in conductivity of PVA films promises about the potential applications in the electrical field.

It is evidenced from the water solubility test and contact angle measurement, that an increase in water contact angle by 85° and large increase in the water solubility time, in comparison with that of pure PVA films. Thus, the shock treated samples shifted from hydrophilic nature to the hydrophobic. This change in PVA films could be associated with the decreased hydroxyl groups in the PVA and increase in carbon content as evidenced by FTIR and EDAX studies. This remarkable shift from hydrophilic to

hydrophobic nature of shock treated PVA films promises the future of PVA for the biodegradable environmental friendly polymer as packaging material.

The gamma irradiation effects revealed that there is a significant increase in the crystallinity and decrease in the conductivity of the PVA samples. Also, through some light on the annealing effect of the shock wave impact effects after gamma irradiation.

5. Important findings of the research work:

- A table top shock tube capable of producing shock waves of Mach numer upto 2 is developed.
- A 'Shock tube Data Processor' device of 5 MHz speed is developed using the open source hardware platform 'Arduino'.
- 'Shock tube Data Analyser' software is developed using the open source software development platform 'PROCESSING'.
- Shock wave treatment on PVA films significantly changes the physical and chemical properties.
- The overall conductivity of the PVA films increases by three times with the shock wave treatment.
- There is a significant increase in the crystalline nature of the PVA samples.
- The hydrophilic nature of PVA films is changed to hydrophobic with the shock wave effect.
- The contact angle of PVA films increased by 85 degrees after shock wave treatment.
- Crystallinity of the shock wave treated PVA films increased significantly after the gamma irradiation.

- There is a decrease in the electrical properties of the shock wave treated PVA samples after the gamma irradiation.

6. Scope for future study

Scope of research in the field of material processing by means of shock wave impacts has no limit; because it is an emerging field of research. The treatment of materials with shock waves as discussed in this work is a novel method of material processing and there is no much work carried out in this field so far. Therefore, the method proposed and used here is an indigenous technique of material processing and the results obtained is also unique and very promising for future developments. In this regard, few extension works immediate to this study are listed below.

- Study of effects of shock waves on PVA films with higher Mach numbers.
- Time bound effects and stability of the changes over time due to shock wave impact..
- Threshold limits of changes in-terms of number of shock wave impacts with respect to the present studies.
- Studies on changes in mechanical properties of shock wave treated PVA films in both continuous impact and increasing magnitude of shock waves.
- Effect of shock waves on the bulk PVA samples etc.

7. List of publications

Journal Papers

- Thirumalesh, S P Raju, K Swaroop and H M Somashekharappa, *"Study on effects of shock wave treatment on PVA films in view of electrical property changes"* 2020 *Mater. Res. Express* **7** 015344, https://doi.org/10.1088/2053-1591/ab6ca1

- Thirumalesh K, Raju SP, Somashekarappa HM, Swaroop K. Shock tube data processing tools using open source hardware and software platforms. *Engineering Reports*. 2020;e12353. https://doi.org/10.1002/eng2.12353

- Thirumalesh , S.P. Raju, H.M. Sosmashekharappa, "*Shock wave treated PVA films as alternative bio degradable polymer for packaging industry" J. Nano- Electron. Phys. 12 No 2, 02034 (2020)*.DOI:https://doi.org/10.21272/jnep.12(2).02034

- Thirumalesh, S P Raju, K Swaroop and H M Somashekharappa, *"Study on effects of gamma irradiation on shock wave treated PVA films"* Polymer Degradation and Stability (Communicated)

- Thirumalesh, S.P. Raju and H.M. Somashekharappa, Swaroop K *"Microstructural changes on PVA films due to Shock waves impacts "* Polymers: - Communicated

Paper presented at National Conferences

- Thirumalesh , S.P. Raju, H.M. Somashekharappa, *"Shock wave impact study on PVA films"* **at 6th National Symposium on Shock Waves-IIT Madras**, Chennai (NSSW-2020), 26th to 28th Feb 2020.

Paper presented at International Conferences

- Thirumalesh , S.P. Raju, H.M. Somashekharappa, "'Construction and optimization of modified Reddy Shock tube using open source platforms', at International conference on Recent Advances in Materials Science and Biophysics (RAMSB 2018), by Department of Studies in Physics Mangalore University, Mangalagangotri-574199. On January 23-25, 2018.

- Thirumalesh , S.P. Raju, H.M. Somashekharappa, *"Study of water resistance property of PVA films with shock wave treatment"*, at INTERNATIONALCONFERENCE ON RECENT ADVANCES IN APPLIED SCIENCES (ICRAAS – 2019), 17th-18th October 2019, Organised by School of Applied Sciences, REVA University, Bangalore.

- Thirumalesh , S.P. Raju, H.M. Somashekharappa, *"Shock wave treated PVA films as alternative bio degradable polymer for packaging industry",* at International Conference on Smart Materials and Nanotechnology (ICSMN-2020), organised by DEPARTMENT OF ENGINEERING PHYSICS & CHEMISTRY SKN SINHGAD COLLEGE OF ENGINEERING, Pandharpur - 413304, Maharashtra, India.

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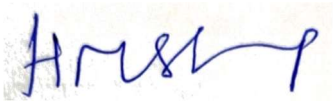
Research Guide



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